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SUMMARY

The vapour deposition process and the properties of an Al-Cr-Fe alloy produced by this process are described. Comparisons are made with some of the rapidly solidified alloys that have been reported.

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1   INTRODUCTION

Rapidly solidified alloys have been under development for many years but few have reached the market place because of the problems of consolidation and the poor properties obtained in some respects. Of particular concern has been the possibility of contamination of the alloy by extraneous material and also by particles of different composition or of different microstructure. The presence of extraneous particles and oxide films can result in low ductility and tough-ness. Consolidation problems are related to the tenacity of the oxide film on aluminium alloys and the ease with which they become hydrated. Avoidance of hydration, or degassing prior to consolidation, is essential if satisfactory ductility and toughness are to be obtained. Very large amounts of deformation are required to break-up and distribute the oxide uniformly throughout the alloy and generally the forging of precompacted billets is insufficient to achieve this. Therefore, extrusion at a high extrusion ratio is often employed to provide material for further mechanical working to shape or as means of producing a near net shape. This can destroy many of the property advantages that are claimed for rapidly solidified alloys due to the high temperatures necessary to achieve a high reduction ratio. An alternative to these techniques is to produce alloys by physical vapour deposition (PVD). A technique for the production of bulk alloys by PVD has been developed at the Royal Aerospace Establishment, Farnborough[1-6]. The advantage of this route is that the alloy is produced as a bulk deposit with only a small amount of mechanical working required to give optimum mechanical properties.

The purpose of this paper is briefly to describe the process options, to describe in more detail the current preferred manufacturing route and to give the properties of an Al-Cr-Fe alloy produced by this process.

2   THE VAPOUR DEPOSITION PROCESS

The process options investigated at RAE have been described elsewhere[1-6] and only a brief description of the methods will be given here. PVD can be achieved in two quite different ways, ie sputtering or thermal evaporation. The rate of evaporation for sputtering is very low, around 100 μm/hour, compared with up to 20 mm/hour for thermal evaporation. Thus thermal evaporation is the obvious choice for bulk alloy production. However, there are various techniques under the general heading of thermal evaporation for the production of vapour deposited alloys. Alloys can be produced by evaporation of the elemental con-stituents of the alloy from separate evaporation baths and condensing them on a moving collector so that the alloy is produced in the form of elemental layers[1]. Such deposits can have high strength, as-deposited or they can be mechanically

worked to produce fairly uniform, dispersion strengthened alloys. Solid solu-
tions cannot be produced by this method, which limits the opportunity for
precipitation hardening but recent research[7] has indicated that ultrafine layered
structures can have very interesting properties.

An alternative technique uses elemental sources for the vapours but the
vapours are allowed to mix before condensing. This technique is unsuitable for
production of bulk aluminium alloys because at high rates of evaporation the
small mean free path in the vapour prevents effective mixing, except at the
boundaries between the vapour streams.

In the current RAE process an alloy vapour is produced by electron beam
heating a liquid alloy melt contained in a ceramic bath, ie co-evaporation.
Electron beam heating is particularly appropriate for the lower vapour pressure
metals such as aluminium and is used because the heat is applied at the liquid
metal surface, ie where the evaporation occurs. The majority of the liquid alloy
runs at a lower temperature, thus saving energy and reducing the erosion of the
crucible, and possibly allowing the use of cheaper refractories. The composition
of the vapour is maintained constant by balancing the rate of evaporation and the
rate of liquid alloy feed. The alloy vapour produced is condensed on a tempera-
ture controlled collector that traverses over the bath so that the deposit builds
up in the form of layers, around 70 μm in thickness, corresponding to a build up
rate, over an area of 500 $\times$ 300 mm, of 6 mm/hour. The deposit surface is mech-
anically worked after each traverse of the bath in order to minimise porosity. A
schematic of the equipment is shown in Fig 1 and a general view, to indicate the
size of the facility, is shown in Fig 2.

The alloy deposit, up to 44 mm thick to date, is sawn from the collector
and machined prior to further mechanical working. Most of these deposits have
been processed to thin sheet by warm rolling.

3   RAE ALLOY 72

Early research at RAE showed that, of the alloy systems that could be
considered for the co-evaporation/co-depositon process, the Al-Cr-Fe system
showed considerable promise, both as a vehicle for the development of the process
and as a useful alloy in itself. A range of alloys were produced with chromium
contents in the range 5 to 9% and iron contents in the range 0.6 to 3.0% giving
tensile strengths up to 865 MPa. Vapour deposition gives the possibility of
producing a range of microstructures, determined by the composition of the vapour
and the deposition temperature. Optimum properties were obtained for RAE
Alloy 72 when deposited at 250°C. In this condition the strengthening is

obtained from three main sources; these are given in Table 1. The three sources contribute equally to strength and the chromium also increases the Young's modulus by 17 GPa. The 'p' phase precipitate shown in Fig.3 is very similar to the precipitates that are present in conventional age-hardened high strength aluminium alloys, where they confer substantial strength but only at low temperature. The 'p' phase precipitates in RAE Alloy 72 are stable to temperatures in excess of 250°C. The vapour deposited alloy can be directly rolled to sheet at temperatures up to 290°C and it is this form, condition A, on which most properties have been determined. The vapour deposited alloy can be directly rolled to sheet at temperatures up to 290°C and it is this form, condition A, on which most properties have been determined.

4   PROPERTIES OF WARM ROLLED ALLOY 72 SHEET (CONDITION A)

4.1   Room temperature strength and modulus

The room temperature tensile properties for 1.6 mm sheet of three alloy compositions are given in Table 2 together with values for a range of competitive alloys[8-14]. Al-7.5%Cr-1.2%Fe is the preferred composition as it gives a good balance of properties but the high strength alloy serves to illustrate the potential of the process. The vapour deposited alloys are denser than conventional aluminium alloys but their specific strength is higher and they can be competitive with the low density aluminium-lithium alloys. Indeed they show significant advantage over the new generation powder metallurgy alloys and Titanium-6Al-4V. Although these properties are very attractive the main advantage of Alloy 72 is in its elevated temperature properties. The last five alloys listed in Table 2 were developed for high temperature applications. The high modulus of RAE Alloy 72 results from the high chromium content and is 20 to 30% higher than for conventional alloys.

4.2   High temperature properties

The thermal stability of Alloy 72 sheet in condition A was assessed by tensile tests at temperatures up to 500°C in the as-rolled condition, Table 3. Strength retention is excellent up to 300°C. Room temperature tests on material heated at temperatures up to 300°C for 1000 hours show that the alloy is stable at temperatures of 250°C and below. Prolonged aging at temperatures above 270°C caused changes in the microstructure; the fine precipitate coarsened initially, followed by inter- and intragranular precipitation of the Al_7Cr phase. This structure provides less resistance to the movement of dislocations and thus reduced room and elevated temperature strength.

The range of aluminium alloys being explored for elevated temperature applications continues to increase but only a few alloy types have achieved commercial status recently. Two alloys of nominal composition Al-8.5%Fe-1.3%V-1.7%Si (FVS0812) and Al-12.4%Fe-1.2%V-2.3%Si (FVS1212) have been developed by Allied-Signal Inc. using planar flow casting, followed by pulverisation and compaction. The properties of these alloys, together with the results from the earlier Al-8%Fe-4%Ce, Al-8%Fe-2%Mo and Al-5%Cr-1.5%Zr-1%Mn alloys from Alcoa, Pratt and Whitney and Alcan, respectively, are given in Fig.4. At temperatures up to 300°C the RAE Alloy 72 is markedly better than the other alloys, particularly when density is taken into account but at higher temperature all of the aluminium alloys tend towards similar strength, with FVS1212 showing a slight advantage.

The creep strength of RAE Alloy 72 is good. Fig.5 compares the creep strength of RAE Alloy 72 (in sheet form) with typical conventional creep resistant aluminium alloys and powder metallurgy alloys Alcoa Al- 8%Fe-7%Ce[15] and Allied-Signal FVS1212. The data for FVS1212 has been extrapolated from data given in the manufacturers data sheet[12].

4.3 Fatigue and fracture toughness

Constant amplitude fatigue tests on RAE Alloy 72 sheet test pieces with a central hole (stress concentration factor K = 2.5) gave a net-section stress of greater than 250 MPa at 10 cycles; the yield strength was 710 MPa. This is much higher than values for conventional fatigue resistant aluminium alloys and also advanced PM alloys such as 7091[9,18]. The rate of growth of long fatigue cracks is similar to that in other strong aluminium alloys. The fatigue crack growth resistance of Alloy 72 is reduced by heat treatment or mechanical working that results in grain boundary precipitation, due to an increased tendency towards grain boundary fracture.

Quenching from the vapour phase can avoid the formation of coarse intermetallics and allow the attainment of higher toughness than can be achieved by ingot metallurgy at this strength level[5]. However, care must be taken to avoid high processing temperatures which can cause grain boundary precipitation of intermetallics, and thus a tendency for intergranular fracture and a decrease in toughness.

4.4 Corrosion resistance of warm rolled sheet

The corrosion resistance of RAE Alloy 72 is exceptional for an aluminium alloy, particularly at this strength level. The weight loss after 6 weeks exposure to 5% sodium chloride fog at 35°C was less than 0.1 mg/cm, ie about

40 times less than for a conventional high strength aluminium alloy. In a standard exfoliation corrosion (EXCO) test the rating was EA (superficial). Fig.6 shows a number of test samples after 96 hours exposure in the EXCO test. The photograph does not do full justice to the RAE 72, which retained its shiny appearance and appeared unaffected.

Stress-corrosion tests were carried out in a constant strain rig; sheet test pieces having tensile strengths in the range 630 to 850 MPa were stressed to 600 MPa and subjected to alternate immersion in neutral 3.5% NaCl solution at 30°C. They survived unbroken after testing for 30 days; there was some superficial blistering but the residual tensile strength was the same as that of unexposed material.

4.5 Formability

The formability of the preferred composition is limited due to the necessity to carry out forming operations at low temperature (*ie* <290°C) but the sheet can, nevertheless, be formed through a 5T bend radius.

5   HEAT TREATED OR HOT WORKED ALLOY 72 (CONDITION B)

Forging or extrusion at low temperature is limited by the high strength of the alloy and, paricularly for forging, its low ductility in the as-deposited condition. Further development of low temperature rolling of plate or low temperature forging must await the availability of larger deposits and suitable working equipment. However, an alternative microstructure is obtained if the alloy is heat treated or mechanically worked at high temperature after deposition, *ie* condition B. Treatment at temperatures greater than around 290°C cause decomposition of the supersaturated solid solution to give a dispersion of intermetallic compound particles in almost pure aluminium. Depending on the time and temperature, intermediate structures can be produced. These structures result in lower strengths than in the cold rolled condition but ductility is increased[16] and the material can be processed more easily, for example by extrusion and forging. Fig 7 shows the microstructure of RAE Alloy 72 extruded at 400°C. The microstructure contains Al_7Cr in a matrix of almost pure aluminium. The high modulus due to chromium is retained even when the chromium is present as the intermetallic compound Al_7Cr[16]. The properties of the alloy in the heat treated or hot worked condition are attractive, particularly as Young's modulus is unaffected by high temperature treatment. Table 4 contains typical room temperature property data and the elevated temperature strength of RAE Alloy 72 condition B extruded at 400°C, using an extrusion ratio of 10:1, is included in Fig 4.

5.1 Corrosion properties of heat treated Alloy 72

Weight loss and EXCO test were also made on Alloy 72 heat treated for
2 hours at 450°C. After 6 weeks exposure to the salt fog already described, the
weight loss was 0.392 mg/cm. This is four times greater than for the warm rolled
sheet but 10 times better than conventional high strength alloys. Exfoliation
was not detected in the EXCO test.

6 OTHER PROPERTIES OF INTEREST

The thermal and electrical conductivity of RAE Alloy 72 in condition A are
15 W/m and 408 respectively, ie similar to titanium or stainless steel. This
results from the very high level of supersaturation of the aluminium lattice by
the chromium in solid solution (>6%). Heat treatment of the alloy at high
temperature, >270°C, causes breakdown of the solid solution and precipitation of
the intermetallic compound Al_7Cr. This causes a rise in electrical and thermal
conductivity to values similar to those for conventional alloys. For example the
electrical conductivity increases from 408 to 82 on heat treatment at 450°C. The
coefficient of thermal expansion is $22.6 \times 10^{-6}/°C$ and the specific heat is
0.218 cal/g°C.

Another unusual property is the high damping capacity of RAE Alloy 72 in
condition B. In condition A, at 14 kHz the internal friction Q = 16 but after
heat-treatment at 450°C (ie condition B) the internal friction increased to
Q = 400. The related damping capacity was measured on much larger samples of
condition B material. Maximum damping occurred at 9 to 12 kHz. High damping
capacity often occurs in metals containing a large number of internal interfaces.
It is probable that the Al_7Cr intermetallic compound particles, that are present
in heat treated material, are responsble. Similar effects are observed in, for
example, cast irons containing graphite flakes and metal matrix composites
containing particulate reinforcement. The high dislocation density in vapour
deposited alloys will also contribute to a high damping capacity.

Rain erosion tests were performed on the RAE Rain Erosion rig and tested
under the standard conditions, ie velocity 233 m/s, rainfield intensity 25 mm/h
and impact angle 90°. After 5 hours exposure the samples showed very light
erosion and pitting. Under the same conditions pure aluminium was deeply eroded
after 1.5 hours exposure, aluminium alloy BS L64 showed light pitting after
5 hours exposure and stainless steel FV120 showed slight pitting after 36 hours
exposure.

A limited number of ballistic impact tests were made on as-deposited RAE
Alloy 72 [18]. The alloy showed a 30% greater resistance to penetration by blunt

kinetic energy projectiles than conventional alloys, Fig 8. The unique micro-structure of the material was mainly responsible for this improvement.

7   MATERIAL COST

The cost of the alloy cannot be accurately estimated. The energy costs associated with evaporation are not large when compared with the total energy content of aluminium alloys but they are an additional cost. Work at RAE on aluminium alloys showed an energy consumption of 6.5 kWh/kg i.. the vapour deposition process. This is only a small addition to total energy consumption for aluminium alloy sheet of 79 to 83 kWh/kg [20]. However, the largest factor is likely to be the cost of the equipment. The cost of the material will then depend on factors such as period over which the equipment is amortised, interest rates, wage rates, output of the equipment etc. An early survey indicated a cost of around £25/kg but a clear idea of the cost of the material will only become apparent when a design study for a commercial plant has been completed. Electron beam technology is now well established. Equipment should become relatively less expensive and result in lower material costs.

8   CONCLUSIONS

The Al-Cr-Fe alloys described here show advances in most properties when compared with both conventional and advanced powder aluminium alloys. The further development of properties and product forms, other than sheet, must await the availability of deposits from a larger scale facility. Research is now concentrated on obtaining further improvements in the properties of aluminium alloys and also the exploration of other alloy systems.

Table 1

THE ROLE OF ALLOYING ADDITIONS AND STRENGTHENING MECHANISMS

Chromium	–	Increases Young's modulus by 2.7 GPa per Wt In solid solution contributes ~⅓ of strength
Iron	–	Present as a 3-5 nm precipitate ('p' phase) Contributes ~⅓ strength
Grain size	– –	Very fine with high dislocation density Contributes ~⅓ strength

Table 2

TENSILE PROPERTIES OF VAPOUR DEPOSITED AND RAPIDLY SOLIDIFIED ALLOYS

Alloy	0.2% Proof stress MPa	Tensile strength MPa	Elong- ation %	Young's modulus GPa	Density Mg/m³	Ref
RAE Alloy 72 A* Al-5.7%Cr-0.7Fe Al-7.5%Cr-1.2%Fe* Al-8.7%Cr-1.5%Fe	607 709 811	616 723 836	6 7.5 7.2	89	2.9	6
RAE Alloy 72B*	510	564	12	89	2.89	
7075-T6 Clad	448	503	8	71	2.79	8
7091-T7E69	505-539	539-582	9.5-14.1		2.82	9
Alcoa Al-8%Fe-7%Ce	460	589	2.4	–	2.95	10, 15
P & W Al-8% Fe-2% Mo-V	393	512	3	–	2.92	11
Allied-Signal FVS0812	387	440	10	88.4	3.02	12
Allied-Signal FVS1212	605	636	8.7	95.5	3.07	12, 15
Alcan Al-5Cr-2Zr-1 Mn	453	474	14	82	2.86	14

* Preferred composition

Table 3

HIGH TEMPERATURE STRENGTH OF RAE ALLOY 72 SHEET

Test Temp °	0.2% proof stress MPa	Tensile strength MPa	Elongation %
ambient	679	719	10.0
200	557	616	1.2
225	232	591	1.3
250	516	544	2.2
275	469	500	1.6
300	428	477	1.5
400	124	187	12.5
450	67	114	26
500	34	56	20

Table 4

ROOM TEMPERATURE PROPERTY DATA FOR RAE ALLOY 72 (CONDITION B)

0.2% proof stress	MPa	510
Tensile strength	MPa	564
Elongation	%	12
Young's modulus	GPa	89

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Direction of collector traverse

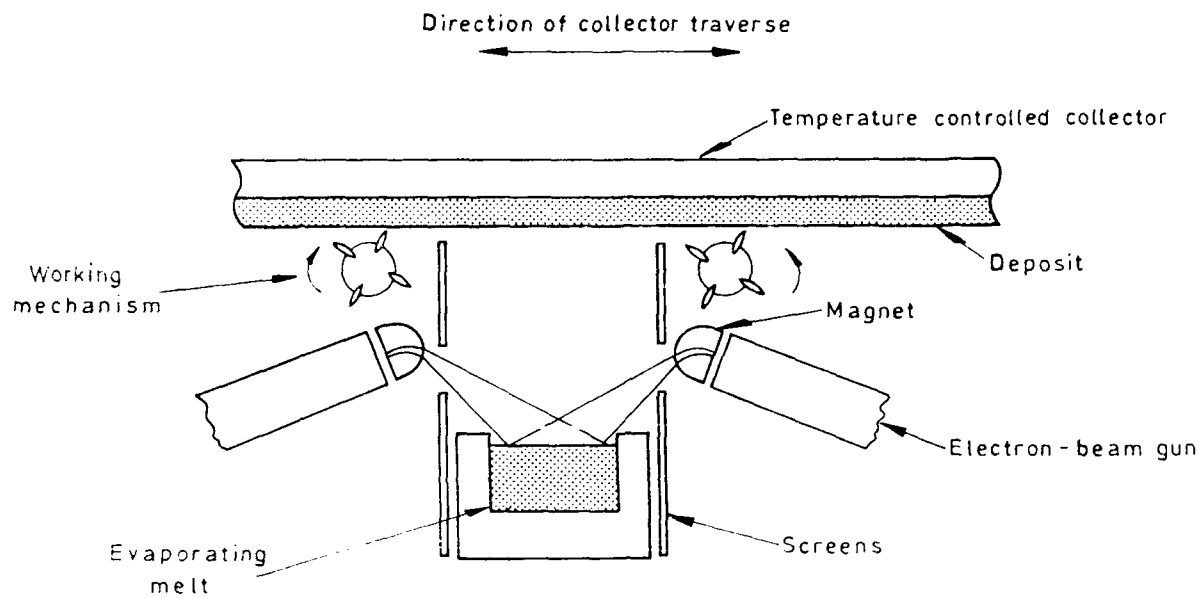

Fig 1   The vapour deposition process

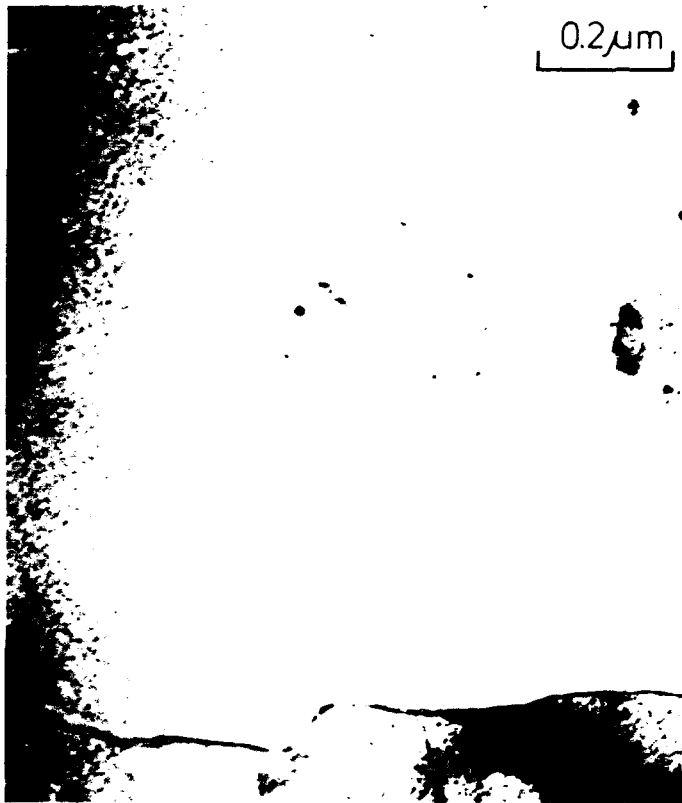Fig 2   General view of the vapour deposition equipment

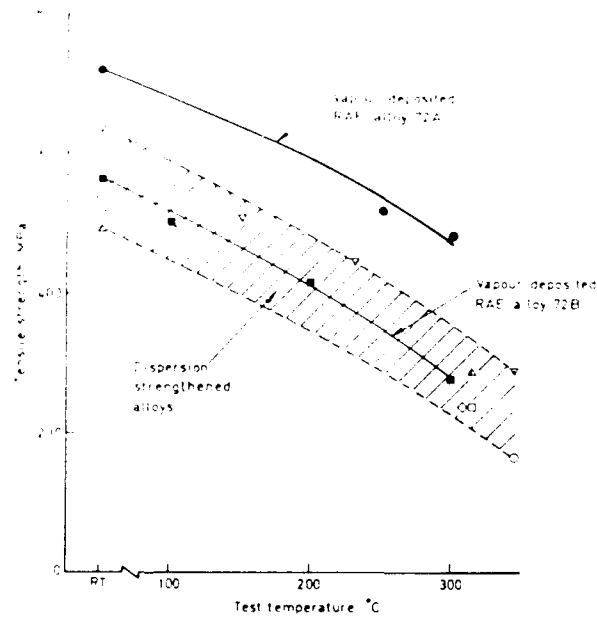Fig 3 Transmission electron micrograph of vapour deposited RAE Alloy 72, as deposited at 170°C and aged at 260°C

Fig 4 High temperature strengths of a range of advanced alloys

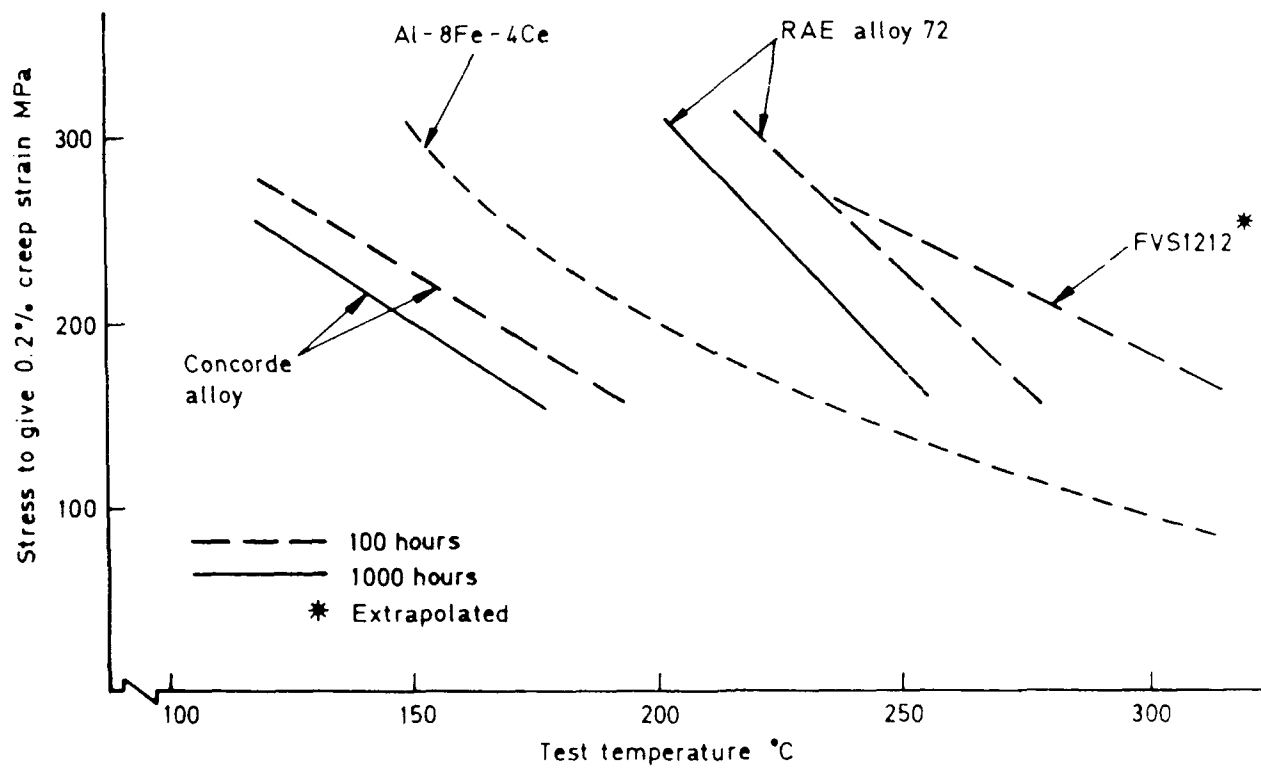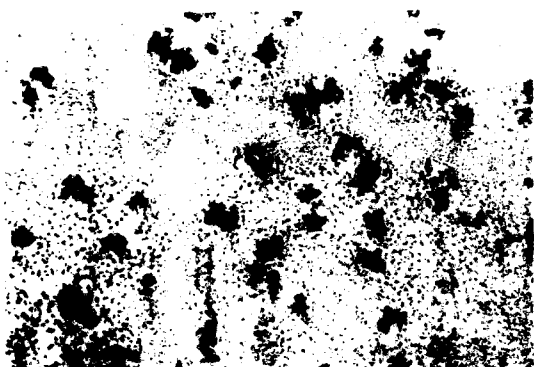| Key	Manufacturer
○ Al - 8Fe - 4Ce	ALCOA
▢ Al - 8Fe - 2Mo-V	Pratt & Whitney
△ Al - 8Fe - 1 5V -1 5Si	Allied - Signal
▽ Al - 12 4Fe - 1 2v - 2 3S₁	Allied - Signal
◇ Al - 4 5 Cr - 1 5Zr-1 2Mn	ALCAN
● Al - 7 5 Cr - 1 2Fe (Condition A)	RAE
▪ Al - 7 5 Cr - 1 2Fe (Condition B)	RAE
▨ Dispersion strengthened alloys	

Fig 5

Fig 5   Creep strength of several aluminium alloys

Fig 6

2024-T3 Chemically de-clad   2024-T3 Clad

L 70 (=L15C)   L 150 (=L157)

**Fig 6   Sheet aluminium alloys after 96 hours
immersion in 'EXCO test solution at 25°C
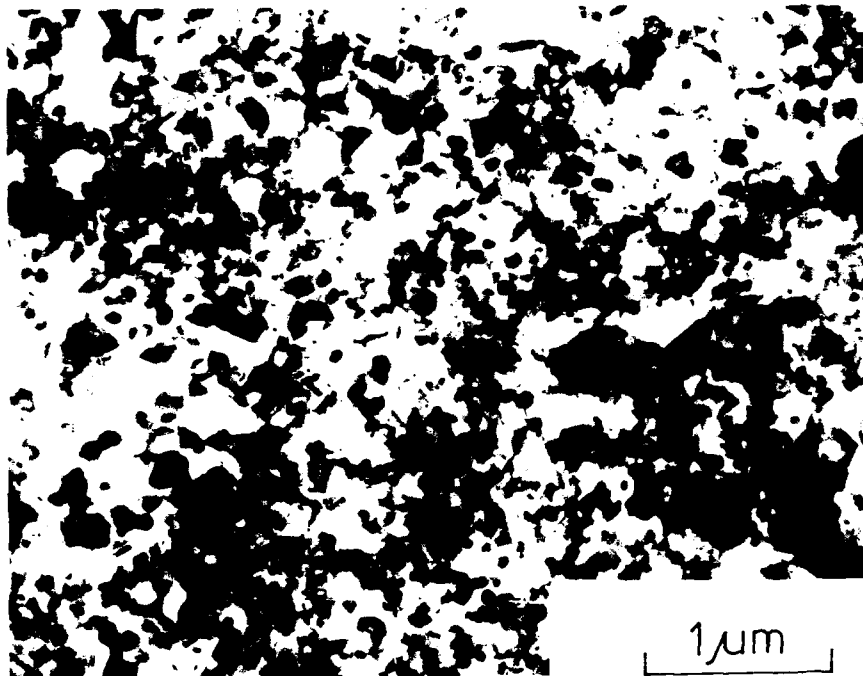(chemically cleaned at conclusion of test)**

Fig 7 Transmission electron micrograph of vapour deposited RAE Alloy 72 extruded at 400°C
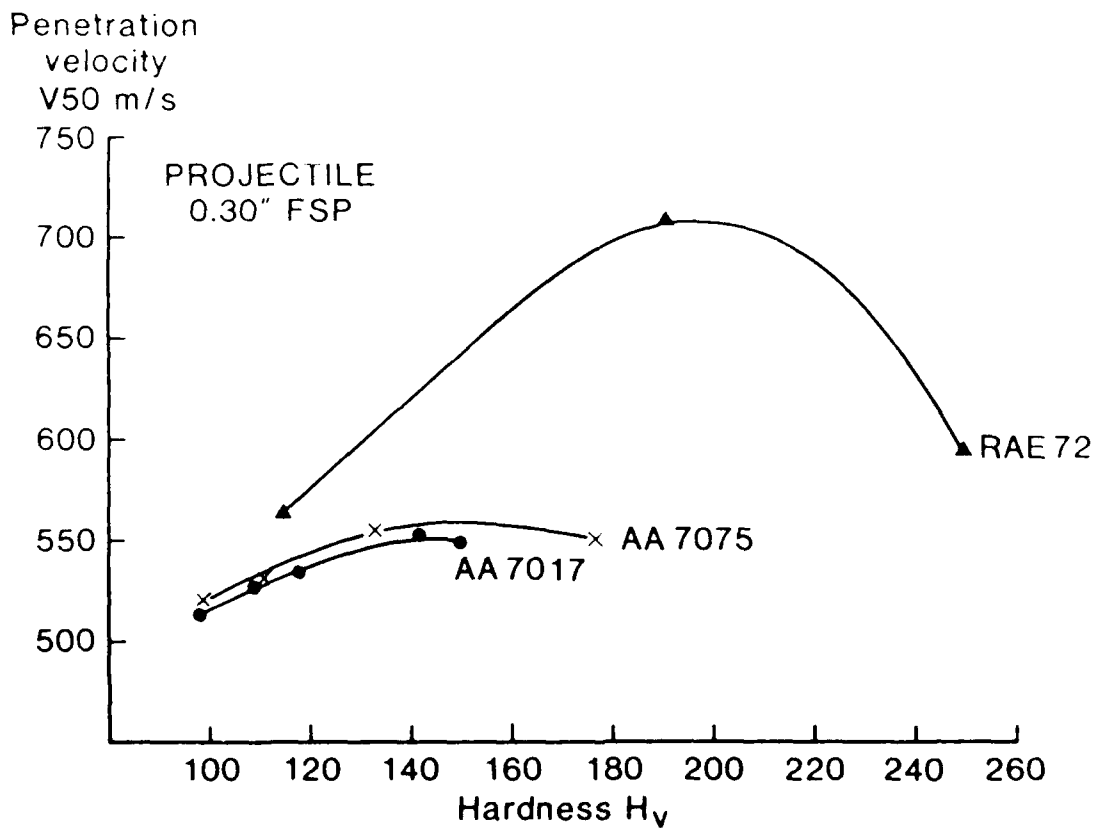

Fig 8 Ballistic impact test results

REPORT DOCUMENTATION PAGE

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17. Abstract

The vapour deposition process and the properties of an Al-Cr-Fe alloy produced by this process are described. Comparisons are made with some of the rapidly solidified alloys that have been reported. Keywords: